PROJECT NUMBER: 1810
PROJECT TITLE: ART Process Development
PROJECT LEADER: R. Prasad
PERIOD COVERED: November, 1989

I. PROJECT ART - COMMERCIAL PLANT SUPPORT

A. **Objective**: To support the Bermuda Hundred Processing Facility startup/operation.

B. **Results**: Support to the commercial plant process optimization continues to be provided. Testing of modified CO_2 fill procedure to minimize bed upsets and CO_2 density fluctuations was initiated at the Commercial Plant. CO_2 flow rates as high as 1400 lbs/min were achieved compared to current flow rate of 1000 lbs/min. Further testing is in progress.

Double batching of stems was tested at the Commercial Plant, i.e., two batches of filler were extracted with a single batch of stems. Extraction targets were met by increasing the amount of stem in the absorber from 1800 lbs dwb to 2100 lbs dwb for the double-batch operation.

Ongoing work at the University of Texas and Pemm Corp, New York, shows the importance of bed packing uniformity and the bed upsets that can occur with fast CO_2 fill and ramp rates. In addition, the design of CO_2 inlet flow distribution system is key in achieving high flow rates without "rat hole" formation or bed bypassing. One-third of the extractor pressure drop should be across the bottom screen to assure uniformity of CO_2 flow distribution. This will require the open area at the bottom screen to be reduced from the current 30% open to about 0.2-1.0% open area. This design change was recommended to the plant management.

C. **Plans**: Continue to support the commercial plant optimization.

II. PROJECT ART - PILOT PLANT

A. **Objective**: To support commercial plant design and flavor development objectives at the Bermuda Hundred Pilot Plant.

B. **Results**: A series of tests at reduced stem OV and/or CPI of CRS were successfully completed to reduce the pressure drop of the absorber. Significant increase in CO_2 flow rate at reduced pressure drop were obtained via reduction of stem OV from 38% OV to 25% OV and/or reduction in stem cuts/inch from 150 cpi to 53 cpi. A combination of stems cuts/inch and OV reduction was implemented at the Commercial Plant, i.e., 76 cpi and 30% OV. Tests are scheduled to increase CO_2 flow rate to design target conditions.

Filler OV reduction (from 28% OV to 22% OV) was successfully tested to reduce the extractor pressure drop and to achieve higher CO_2 flow rates. However, the filler was underextracted due to the low AB level used. Development of ways to apply ammonia and/or alternative bases has been initiated with a target filler OV of 21%.

Batch water column trials were started with appropriate safety protocols in place. Initial testing with acid water was completed (without filler) to determine appropriate operating conditions. A CO_2 flow rate of 267 lbs/min resulted in no carry-over of acid water solution from the absorber into the pipes. A run with pre-extracted filler was completed to simulate full operating conditions. Training of operating personnel in full safety gear was done, along with sampling of nicotine in air. As expected, the filler increased in moisture content from 25% OV to about 30-35% OV. The nicotine content of pre-extracted DL blend filler was reduced from 0.15% to 0.09% dwb using acid water in the baskets.

The first batch water column run was successfully completed. DL blend filler was extracted from 2.73% nicotine to 0.07% nicotine level (97.3% extraction) at 2000 psi, 140°F, 200 M/M. No carryover of acid water from the absorber to the pipes took place at 267 lbs/min CO_2 flow rate. However, due to an unexpected "stop cycle" during CO_2 recovery step, backflow of acid water to the lower baskets was experienced. The nicotine from CO_2 was absorbed in the bottom two baskets, indicating excellent entrapment capabilities of acid water as well as excellent efficiency of contact between CO_2 and water provided by the basket sieve tray design. As expected, the filler OV increased (from 25.8% initially to 28.8% "fluffed").

Loss of non-nicotine volatiles, "flavors", was measured by GC in the water effluent from the batch absorber. Total loss of volatile flavor, compared to the amount of nicotine removed, was in the range of 0.02 to 0.07 parts per one part nicotine. Subjective testing of extracted filler is in progress.

A safety review of pilot plant procedures was completed to determine the feasibility of running DI water in the batch water column. The required safety protocols to protect from nicotine in air were outlined. A limited number of runs with DI water are possible at BHPP provided the new safety protocols are followed. The expected filler subjectives should be equivalent to the proposed interim water column "pipe". This negates the need for a 12 inch pipe at an additional cost of $250M to $500M in the 650 being prepared by PM Engineering.

C. **Plans**: Test the extractor pressure drop reduction at reduced OV and at adequate AB level. Implement the finding in the Commercial Plant to achieve capacity increase.

Explore process alternatives to improve the product attributes.

III. PROJECT ART - NEW PROCESS DEVELOPMENT

A. **Objective**: To develop new processes for scale-up and implementation in the Bermuda Hundred Facility.

B. **Results**: A preliminary scope of separations pilot plant using clay systems was prepared using fluidized bed concept in the upflow mode of operation. Further discussions with designers from clay equipment vendors indicated that upflow fluidized bed design is not practiced in the industry and a plate and frame filter is the preferred option.

A literature survey of the clay applications in the industry was initiated. The measurement of key process parameters for the design of pilot scale system was initiated, i.e., settling velocity, fluidization velocity, particle size distribution vs pressure drop.

A 650 for continuous water column is being prepared by PM Engineering. Plans are to install the cooling heat exchanger in February 1990 and to install the continuous water column by September 1990.

C. **Plans**: Continue laboratory and pilot scale development activity on selective separation of nicotine from flavor.